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Pulps — Preparation of laboratory sheets for physical testing —

Part 2: **Rapid-Köthen method**

Pâtes — Préparation des feuilles de laboratoire pour essais physiques —

Partie 2: Méthode Rapid-Köthen



Reference number ISO 5269-2:2004(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 5269-2 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*, Subcommittee SC 5, *Test methods and quality specifications for pulps*.

This third edition cancels and replaces the second edition (ISO 5269-2:1998), of which the Introduction, Clauses 1, 2 and 7 and Subclause 5.1 have been revised.

ISO 5269 consists of the following parts, under the general title *Pulps* — *Preparation of laboratory sheets for physical testing*:

- Part 1: Conventional sheet-former method
- Part 2: Rapid-Köthen method

Introduction

It has been agreed that the ultimate aim of standardization of the preparation of laboratory sheets should be to develop one method which is internationally acceptable and which, if possible, permits the use of different types of sheet-making apparatus.

For practical reasons, it has not proved possible to achieve this at present. Therefore, as an interim measure, in view of the widespread use of equipment described in this part of ISO 5269, it has been decided to provide agreed guidance on the use of different types of equipment in order to achieve consistency of results with each method.

To avoid creating too many levels of results, the method specified in this part of ISO 5269 should preferably be used with the PFI mill method of laboratory beating according to ISO 5264-2. The method specified in ISO 5269-1 (Conventional sheet-former method) should preferably be used with the Valley beater or PFI mill methods of laboratory beating according to ISO 5264-1^[2] and 5264-2, respectively.

Pulps — Preparation of laboratory sheets for physical testing —

Part 2:

Rapid-Köthen method

1 Scope

This part of ISO 5269 specifies a method, using a Rapid-Köthen sheet former, for the preparation of laboratory sheets of pulp for the purpose of carrying out subsequent physical tests on these sheets in order to assess the relevant properties of the pulp itself.

This part of ISO 5269 is applicable to most kinds of pulp. It is not suitable for some pulps with very long fibres, such as those made from unshortened cotton, flax and similar materials.

This method is not suitable for the preparation of laboratory sheets for the determination of diffuse blue reflectance factor (ISO brightness) in accordance with ISO 3688^[1].

WARNING — When long-fibred pulp is used in the unshortened form, the sheet formation may not always be satisfactory.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 187, Paper, board and pulps — Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples

ISO 4119, Pulps — Determination of stock concentration

ISO 5263-1, Pulps — Laboratory wet disintegration — Part 1: Disintegration of chemical pulps

ISO 5263-2, Pulps — Laboratory wet disintegration — Part 2: Disintegration of mechanical pulps at 20 °C

ISO 5263-3, Pulps — Laboratory wet disintegration — Part 3: Disintegration of mechanical pulps at ≥ 85 °C

ISO 5264-2, Pulps — Laboratory beating — Part 2: PFI mill method

ISO 5269-1, Pulps — Preparation of laboratory sheets for physical testing — Part 1: Conventional sheet-former method

3 Principle

A circular sheet is formed from a pulp suspension on a wire screen under suction. The sheet is subjected to pressure and dried in a dryer, with almost complete prevention of shrinkage, in a specified way with respect to the pressure applied, the suction and the temperature.

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Equipment 4

- Rapid-Köthen apparatus, consisting of a sheet former, transfer equipment, one or more dryers and accessories (see Figure 1). The parts of the instrument which come into contact with stock or water shall be corrosion resistant.
- Sheet former, consisting of a stock container for preparing the pulp suspension, the screen section on which wet fibre is forwarded, and the suction chamber for drawing off and holding the water after passing through the screen. The lower part of the stock container, screen section and suction chamber may be sealed off

4.1.1.1 Stock container (see Figure 1)

The stock container consists of a transparent cylindrical tube (1) with an internal diameter of 200 mm ± 0,5 mm and a capacity of not less than 10 litres. The container is graduated in litres. Around the circumference of the lower part of the stock container are two rows of holes (2), 1,5 mm in diameter, one above the other, connecting radially to a circular cavity (3) round the outside of the cylinder. Each row contains 40 holes. The distance between the screen surface and the lower row of holes is 10 mm, and the space between the lower and upper rows is 7 mm. The lower row of holes is drilled horizontally and the upper row is inclined upwards at an angle of 30° towards the centre of the cylinder.

The stock container is so constructed that it can be lifted off the screen section. The circular cavity can be filled with water and compressed air.

4.1.1.2 Screen section

The screen section, which separates the stock container and the suction chamber, consists of the sheetforming screen (4) and the supporting screen (5).

The nickel sheet-forming screen gauze is evenly stretched over the ring and can be lifted off from the supporting screen. The dimensions of the twilled sheet-forming screen gauze are as follows:

Number of warp wires: 60 wires/cm Number of weft wires: 55 wires/cm

Wire diameter: 0.060 mm to 0.065 mm

The supporting screen gauze is evenly stretched over bars (6) having a width of 2 mm and a height of 30 mm arranged parallel to one another and spaced 10 mm ± 2 mm apart. The two upper edges of each bar are chamfered at an angle of 75° to the horizontal in such a way that a 0,5 mm wide ledge remains as a support for the supporting screen. The supporting screen shall be exactly horizontally aligned.

The dimensions of the supporting screen gauze in simple plain weave, made from phosphor bronze are as follows:

Number of warp wires: 8 wires/cm Number of weft wires: 7 wires/cm Wire diameter: 0,35 mm

4.1.1.3 Suction chamber

The suction chamber has a capacity of more than 10 litres, and has a water outlet (7) that can be closed. The suction chamber can be connected to a vacuum pump by means of a suction tube (8) placed at its axis, and covered so that it is protected against the infiltration of water. A regulating vent limits the maximum suction in the suction chamber to 27 kPa.

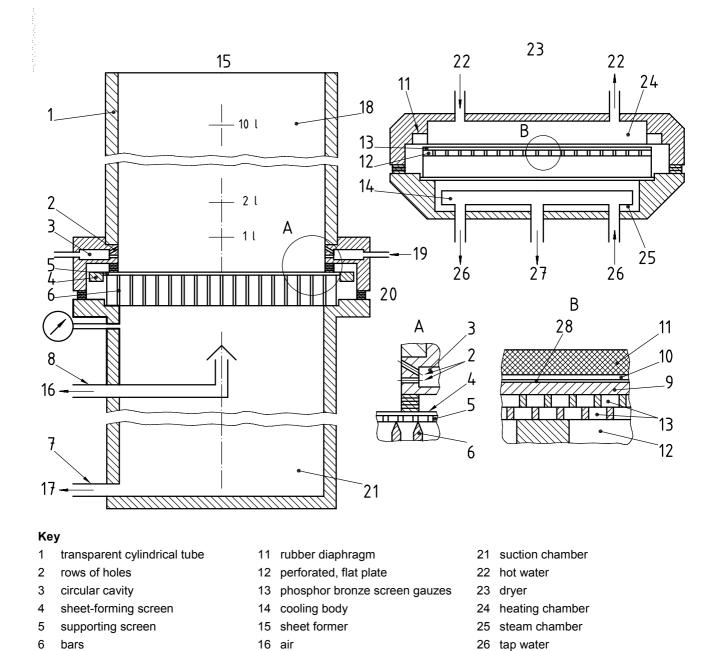


Figure 1 — Rapid-Köthen sheet-forming and drying instrument

27

vacuum pump

laboratory sheet

17

water

19 water or air

18 stock container

20 screen section

4.1.2 Transfer equipment for the wet fibre sheet, consisting of a couch roll, carrier boards and paper cover sheets.

4.1.2.1 Couch roll

water outlet

suction tube

carrier boards

10 paper cover sheets

7

8

9

The couch roll has a diameter of 120 mm to 130 mm, a length of 240 mm to 260 mm and a mass of 3,0 kg \pm 0,2 kg. The outer surface shall be resilient and is preferably made of felt about 20 mm thick.

4.1.2.2 Carrier boards (9)

Circular sheets of bleached lined folding boxboard, without colorants and fluorescent whitening agents, having a diameter of 240 mm, a grammage of 200 g/m², and a well-sized surface.¹⁾

4.1.2.3 Paper cover sheets (10)

Circular sheets of well-sized calendered writing paper, with a grammage of 65 g/m² \pm 5 g/m², and a diameter of 205 mm.¹⁾

Dryer, consisting of a heating chamber with a rubber diaphragm to transmit the heat and pressure, the supporting screen and a steam chamber fitted with a cooling body. The heating chamber, supporting screen and steam chamber can be sealed off.

Heating chamber 4.1.3.1

The chamber consists of a cavity provided with an inlet and an outlet for hot water at 93 °C ± 4 °C. The bottom of the cavity consists of a temperature-resistant, highly elastic rubber diaphragm (11) of thickness 1 mm to 2 mm. The diameter of the rubber is slightly greater than 201 mm. The heating chamber can be lifted off the supporting screen. With a submerged heating chamber, the distance between the flat rubber diaphragm and the supporting screen is 1 mm.

4.1.3.2 Supporting screen

The screen consists of a perforated, flat plate (12), above which two phosphor bronze screen gauzes (13) are stretched and firmly attached to the edge of the plate. The plate shall be so supported that negligible deformation occurs under a surface pressure of 100 kPa.

The dimensions of the supporting screen are as follows:

Upper screen gauze

Number of warp wires: 32 wires/cm Number of weft wires: 24 wires/cm

Wire diameter: 0,16 mm to 0,17 mm

Lower screen gauze

Number of warp wires: 8 wires/cm Number of weft wires: 7 wires/cm Wire diameter: 0,35 mm

Supporting plate

Thickness: 2 mm

Hole diameter: 3 mm to 4 mm

Spacing of holes: 5 mm

Suitable carrier boards and paper cover sheets are available commercially.

¹⁾ Further properties of carrier boards and paper cover sheets that have proved suitable are given below.

Carrier boards made of imitation chromoboard, based upon mechanical pulp covered with woodfree, machine-sized white top liner. The grammage of top liner is $60 \text{ g/m}^2 \pm 5 \text{ g/m}^2$, the Cobb water absorption is $20 \text{ g/m}^2 \pm 5 \text{ g/m}^2$ and the Bekk smoothness is 20 s to 25 s. The air permeance of the carrier board should exceed 1.83×10^{-6} m/Pa·s, and the apparent density is 0,65 g/cm³ to 0,70 g/cm³.

Paper cover sheets of woodfree, supercalendered white paper, with a mass fraction of ash less than 5 % with a Cobb water absorption of 15 g/m 2 \pm 2 g/m 2 , and a Bekk smoothness of 80 s to 120 s.

4.1.3.3 Steam chamber

The chamber consists of a hermetically sealed cavity situated directly below the supporting screen, and having an exhaust nozzle at the deepest position. The steam chamber shall be evacuated by the exhaust nozzle to a pressure of 5 kPa absolute.

A cooling body (14) with a large surface, through which tap water flows, is fitted in the steam chamber. The temperature of the cooling water shall not exceed 20 °C. Part of the steam passing through the supporting screen condenses on the cooling body.

4.2 Accessories

The following are important accessories of the Rapid-Köthen sheet forming and drying instrument, apart from the measuring equipment for checking compliance with the above-mentioned operating conditions.

4.2.1 Dilution water pump, for passing water at (18 to 20) litres/min into the stock container (4.1.1.1) of the sheet former.

NOTE The same pump is generally used for the passage of dilution water, cooling water and compressed air, as well as for evacuation.

- **4.2.2 Compressed air pump**, for passing air at about 60 litres/min into the stock container (4.1.1.1) of the sheet former.
- **4.2.3** Hot-water heater, for providing heating water at a temperature of 93 $^{\circ}$ C \pm 4 $^{\circ}$ C.
- **4.2.4 Hot-water pump**, for circulating the heating water between the hot-water heater and the heating chamber (4.1.3.1) at a delivery rate of (3 to 6) litres/min per heating chamber.
- **4.2.5 Cooling water pump**, for passing tap water at about 2 litres/min through the cooling body in the steam chamber (4.1.3.3) of the dryer.
- **4.2.6 Vacuum pump**, capable of evacuating the steam chamber (4.1.3.3) of the dryer within 10 s to a pressure of 5 kPa absolute.

5 Pretreatment and preparation of sample

5.1 Pretreatment

5.1.1 Unbeaten pulps

Disintegrate the pulp in accordance with the three parts of ISO 5263. If the sample is a chemical pulp, disintegrate in accordance with ISO 5263-1, if the sample is a mechanical pulp not exhibiting any latency, disintegrate in accordance with ISO 5263-2, and if the sample is a mechanical pulp that exhibits latency, disintegrate in accordance with ISO 5263-3.

5.1.2 Laboratory-beaten pulps

Prepare the pulp as specified in ISO 5264-2.

5.1.3 Slush pulps taken from the production line

No pretreatment is necessary.

Preparation of sample

Dilute the stock to a mass fraction of 0,2 % to 0,5 %. For pulps that tend to produce flocs, dilute the stock to a mass fraction of 0,2 % to 0,3 %. Determine the stock concentration (mass fraction) in accordance with ISO 4119, or make a laboratory sheet (oven-dry grammage 65 g/m² to 85 g/m²) of known area, as specified in 6.1. The determination of the mass fraction of the stock, with the aid of this laboratory sheet, eliminates the need for making a correction for the fines loss through the sheet-forming screen (see the third paragraph in 6.1).

The diluted stock shall be used for sheet making with the least possible delay.

Procedure

Sheet forming

Place the cleaned sheet-forming screen on the supporting screen (see 4.1.1.2). Place the stock container (4.1.1.1) on the screen section (4.1.1.2), and close the outlet opening of the suction chamber (4.1.1.3).

Run 4 litres of tap water into the stock container using the dilution water pump (4.2.1). Pour into the top of the stock container a quantity of stock, prepared as specified in 5.2, corresponding to 2,4 g of oven-dry pulp and to a grammage of the ready-made sheet of 75 g/m² ± 2 g/m². Add more dilution water to the stock container, until it contains a total of 7 litres. If the sheets are used for the determination of certain properties such as stiffness and compressive properties, the grammage of the sheet shall be 140 g/m² ± 4 g/m², calculated on an oven-dry basis.

In most instances, fines loss occurs, which shall be compensated by the addition of extra pulp.

Immediately pass air into the stock container by means of the compressed air pump (4.2.2) for a period of 5 s to agitate the stock.

Switch off the compressed air supply and allow 5 s for the turbulence to cease. Start dewatering by opening the valve connecting the vacuum pump (4.2.6) to the suction chamber.

During the dewatering, the suction shall not exceed 27 kPa (see also 4.1.1.3).

From the moment all of the suspension water has been sucked through the fibre sheet, draw air through the sheet by suction for a further 10 s ± 1 s. Switch the vacuum pump off, release the vacuum in the suction chamber, and open the water outlet of the suction chamber. Remove the stock container from the screen section.

NOTE The formation of sheets from pulps that tend to be flocculent can be improved by accelerated dewatering. This can be done by opening an outlet of the suction chamber to the atmosphere (not shown in Figure 1) for about 2 s at the beginning of the dewatering.

CAUTION — If the dewatering is controlled by an automatic timer, a significant volume of suspension may remain in the stock container when the process is terminated. This feature has been observed when sheets have been prepared from groundwood or short-fibre pulp.

6.2 Transfer of the sheet

Place a carrier board (4.1.2.2) with its smooth side downwards and a paper cover sheet (4.1.2.3) in the centre of the wet sheet. Roll the couch roll (4.1.2.1) back and forth over the sheet over a 2 s period without applying additional pressure, once in each of two directions at right angles to each other. The couch roll shall be placed on the edge of the screen but not on the wet sheet.

The carrier board and paper cover shall only be used once.

Remove the sheet-forming screen together with the wet sheet and carrier board from the supporting screen (4.1.3.2) and lay it slightly inclined to the horizontal but with the edge against a horizontal underlay, releasing the carrier board to which the wet sheet is attached onto the receiving underlay.

Before using the sheet-forming screen again, clean off any adhering fibres with a strong jet of water applied from the back of the screen.

6.3 Drying and conditioning

Within 1 min of removing it from the screen, place the wet sheet, attached to the carrier board (4.1.2.2) on the supporting screen (4.1.3.2) of the opened dryer. Place a paper cover (4.1.2.3) on the wet sheet, close the dryer immediately, and evacuate by means of the vacuum pump (4.2.6) (see paragraph 2 in 6.2).

The drying period, calculated from the time of reaching the prescribed suction of 96 kPa, is about 5 min to 7 min. Switch the vacuum pump off, release the vacuum in the dryer, and remove the finished sheet with carrier board and cover paper. Remove the cover paper and carrier board from the finished sheet.

A prolonged drying period (minimum 12 min) is necessary if sheets of high grammage are made.

Before testing, condition the Rapid-Köthen laboratory sheets in accordance with ISO 187.

7 Test report

The test report shall include the following information:

- a) reference to this part of ISO 5269;
- b) all information necessary for complete identification of the sample;
- c) statement of the method of sampling;
- d) the disintegration procedure used (ISO 5263-1, ISO 5263-2 or ISO 5263-3);
- e) the target grammage of the sheets;
- f) characteristics of the water used (such as pH, hardness);
- g) any unusual features observed in the course of the procedure;
- h) any operations not specified in this part of ISO 5269 or in the International Standards to which reference is made, or regarded as optional, which may have affected the results.

Bibliography

- ISO 3688:1999, Pulps Preparation of laboratory sheets for the measurement of diffuse blue [1] reflectance factor (ISO brightness)
- [2] ISO 5264-1:1979, Pulps — Laboratory beating — Part 1: Valley beater method

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